AD-A272 657

WL-TR-93-4032

A STUDY OF THE INDUCTION HEATING OF ORGANIC COMPOSITES

G. William Lawless
University of Dayton Research Institute
300 College Park Avenue
Dayton, OH 45469-0138

Ted Reinhart
Materials Directorate
Wright Laboratory
Wright-Patterson AFB, OH 45433-7734

FEBRUARY 1993

Interim Report for the Period October 1989 - June 1992

Approved for public release; distribution is unlimited.

MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-7734

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility nor any obligation whatsoever. The fact that the government may have formulated, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise in any manner construed, as licensing the holder or any other person or corporation, or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

ROBERT B. URZI
Materials Engineering Branch
Systems Support Division
Materials Directorate

THEODORE J. REINHART
Chief, Materials Engineering Branch
Systems Support Division
Materials Directorate

THOMAS D. COOPER
Chief, Systems Support Division
Materials Directorate

If your address has changed, if you wish to be removed from our mailing list, or if the addressee is no longer employed by your organization, please notify WL/MLSE, Wright-Patterson AFB, OH 45433-7718 to help us maintain a current mailing list.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
February 1993

3. REPORT TYPE AND DATES COVERED
Interim; October 1989-June 1992

4. TITLE AND SUBTITLE
A STUDY OF THE INDUCTION HEATING OF ORGANIC COMPOSITES

5. FUNDING NUMBERS
C-F33615-89-C-5643
PE-62102
PR-2418
TA-04
WU-69

6. AUTHOR(S)
G. William Lawless
Ted Reinhart

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
University of Dayton Res. Inst., 300 College Park Ave.,
Dayton, OH 45469-0138
Wright Laboratories, Materials Directorate,
Wright-Patterson AFB, OH 45433-7734

8. PERFORMING ORGANIZATION REPORT NUMBER
UDR-TR-92-146

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Materials Directorate
Wright Laboratory
Air Force Materiel Command
Wright-Patterson Air Force Base, OH 45433-7734

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
WL-TR-93-4032

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

A study of the application of induction heating to graphite-based composites, both thermoset and thermoplastic. Panels of IM-6/3501-6 and IM-6/PEEK were processed in magnetic fields of 450-kHz and 25-kHz frequencies. A 15.24-cm-diameter pancake-type induction coil was used with 10.2-cm x 10.2-cm, 16-ply specimens. Vacuum bagging provided the only consolidation pressure. Data include resin, fiber, void contents, 3-pt. and 4-pt. loading for mechanical properties evaluation, and thermal analysis (DSC and TMA). Comparisons are made to similar samples processed under a standard autoclave cycle. Additional variables studied and discussed include the use of various caul plate materials as field susceptors, effects of coil vibration, various prepreg lay-up orientations, and other sample thicknesses. A brief discussion of the overall field effects and value of induction heating for field repair is included.

14. SUBJECT TERMS
Composites; Vacuum Bagging; Induction Heating; Processing

15. NUMBER OF PAGES
35

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UNLIMITED

PREFACE

This technical report was prepared by Ted J. Reinhart of the USAF Wright Aeronautical Laboratory, Materials Directorate (Wright-Patterson AFB, OH), and G. Wm. Lawless of the University of Dayton Research Institute (Dayton, OH). The work for this effort was performed at the Materials Directorate during the period from October 1989 to June 1992 under Contract No. F33615-89-C-5643. The authors wish to thank the following individuals who contributed substantially to this effort: Robert Askins, Susan Saliba, Sam Macy, Dr. Perry Yaney, and Dr. Richard Harmer, all of the University of Dayton Research Institute.

We express our appreciation to Robert Askins of the University of Dayton Research Institute for his design and suggestions of this test panel layout, also to Susan Saliba for her continuing cooperation and support in obtaining the physical and mechanical properties data.

This report was submitted by the authors in February 1993.

TABLE OF CONTENTS

LIST OF TABLES

SECTION 1
INTRODUCTION AND BACKGROUND

The use of a rapidly oscillating magnetic field, i.e. induction heating, for the processing, heat treatment, and handling of metals has been recognized as a versatile and effective tool for over a century. This "electrotechnology," as applied to metals, comprises a well-defined and large-scale industry. The primary mechanism of heat generation in the metal is attributed to the I^2R loss from induced eddy currents, the so-called Joule effect. The eddy currents circulate in phase with the inducing field in a plane perpendicular to the field, or parallel to the coil, and like all currents, they require a complete electrical pathway. They flow predominantly on the surface of the metal. The heating effect is, therefore, reduced with specimen thickness, and the terms "effective depth" or "skin effect" are often used in a descriptive sense. The degree of meaning here is dependent upon several variables: power level or field intensity, field frequency, distance of separation between coil and sample, overall efficiencies, and certain material properties. Material electrical resistivity (or its reciprocal, the conductivity), and its magnetic permeability are especially relevant properties. We must consider the sample material as an element of the overall electrical circuit. It forms a "load" on the power supply.

The theory behind induction heating is usually based upon an analysis of a solid rod specimen inside a cylindrical, i.e., Helmholtz, coil (1). This represents the simplest geometry in a well known and reasonably uniform field.

Ferromagnetic materials below their Curie point gain an additional source of heat due to the hysteresis loss from the rotation of their magnetic domains, again in phase with the inducing field. This is a type of molecular friction, but the Joule loss is the dominant mechanism for all conductive materials whether magnetic or not (1,2).

We must note that this is a well founded industry, both commerically and scientifically. Its "scale" might be gauged by noting that an American company has an operational induction coil over 18 m in diameter and over 49 m high, and that experiments on board a recent space shuttle used induction heating for the production and purification of special alloys in the weightless and magnetic free environment of outer space.

More recently this technology has been transferred to the field of organic composites (3,4,5). There are two principle motivations here; one to gain processing advantages with time and economics and the other for repair. The Systems Support Division of the Materials Directorate, Wright Laboratory (the sponsor of this effort) has the field repair of aircraft as one of its major responsibilities. The increasing use of advanced composites on aircraft mandates the development of advanced field repair techniques. In addition, while most present day operational composite systems are processed, and repaired at 177°C, future systems will no doubt require higher temperatures, well within the range of induction heating. Field repair also brings limitations of available power (120-V / 20-amp circuitry are generally all that are available), and further requirements of portability, simplicity of operation, localized and controlled heating, nondamage to adjacent components, and vacuum bagging which is the only available consolidation pressure.

The purpose of this work was to evaluate the potential of induction heating as an alternative method for the successful application of an aircraft patch in the field. This will entail surface/area preparation, adhesive bonding, and the application of a precured, or in situ cured composite patch. The processing of the "patch" became the focus of this effort; overall, the work was as much educational as it was applied.

The organic composite is very unlike a solid uniform mass of conductive metal. There is no magnetism and only the carbon fibers are electrically conductive. Furthermore the conductivity of carbon is unlike that of any metal; its value is much less than that of common metals, but it increases with temperature, contrary to that of common metals. An approximate comparison to steel shows that at room temperature a typical magnetic steel is over 200 times as conductive as carbon, but at 538°C, only 30 times. This point is made because if the mechanism of heat generation from induction heating in a composite is due to the conductivity of the carbon fibers, it may become important to consider this rather precipitous change with temperature. In this regard, we might note that the fibers are also insulated one from the other, more or less in a given ply, and probably quite effectively from ply to ply.

The size of the fiber is also of fundamental importance. It is too small for any practical consideration of surface versus in-depth heating; the "skin effect," and its small size mandates a high frequency field to induce coupling. But to incorporate portability into field repair, lower frequency generators, at least at present, must be used. This admits the possibility of heating the prepreg indirectly, as with a susceptor. In other

words, either the composite prepreg will couple, and therefore heat directly with the applied field, or a susceptor, for example, a metallic caul plate, can be used. The lower frequency magnetic flux will create eddy currents in the susceptor, and the resultant heat can be more than enough to melt even a thermoplastic prepreg quite easily. Using the susceptor as a caul plate serves as double duty.

Until recently the mechanism of inductively heating an organic composite was believed due to the Joule heat loss from eddy currents being generated along the carbon fibers. Electrical contact was demonstrated or assumed. It was the most obvious explanation and it was easily shown that crossed plies were necessary to trigger the mechanism, thereby adding further confirmation. But Gillespie (5 through 9) has recently published a series of papers that point to dielectric loss of the matrix as the dominant mechanism. While it was not the nature of the present work to explore the fundamental mechanism of the energy transfer, the results of one particular experiment may be significant toward that end.

A $(0,90)_{4S}$ 10.2-cm x 10.2-cm thermoplastic composite laminate was assembled with each ply insulated one from the other, but in two different ways. Sheets of 0.051-mm Kapton (DuPont polyimide film) were cut in squares, 7.62 cm x 7.62 cm, in the one case to insulate the center portion of the plies. In the other instance, the Kapton sheets were cut to only insulate the outer 1-centimeter margin around each composite ply. Together these methods of insulation constitute a picture frame technique: in one case the picture was insulated, in the other case, only the frame. In both cases, the layup was vacuum bagged, positioned close to the inductive coil, and power applied. Both power generation units as described herein were used. The picture-insulated layup heated, the frame-insulated layup did not. There is no intent here to quantify any theory, only to point out that under the conditions tested, ply-to-ply contact around the perimeter is clearly essential for composite heating.

Field frequencies of 450 kHz and approximately 25 kHz were used. The higher frequency excited the composite samples directly, and the 25 kHz, obtained from a portable low-power source, when used with a proper caul plate susceptor, also generated sufficient heat for processing. This point will be given further elaboration.

The work of Gillespie, in addition to that of Border (3,10), Miller (4,11) and others (12) points to the growing interest of applying this technology to composite materials.

SECTION 2
EXPERIMENTAL

Two composite systems were studied: IM-6/3501-6 and IM-6/PEEK. Their standard autoclave cure cycles as used herein are shown in Table 1. The specimen size was usually 10.2 cm x 10.2 cm, 16 ply. The lamina orientations were varied.

TABLE 1
STANDARD AUTOCLAVE PROCESSING CYCLES

IM-6/3501-6	IM-6/PEEK
Bagged layup under full vacuum	Bagged layup under full vacuum
Heat from room temperature	Heat from room temperature
1-3°C/m Pressure to 483kPa	Pressure to 110kPa 4-5°C/m
Vacuum released when P = 483kPa	Vacuum maintained throughout
Hold 1 hr. at 116°C	Pressure to 414kPa when T = 224°C
1-3°C/m to 179°C; Hold 2 hrs.	Hold 30-45 min. at 382°C
Controlled cool to 65°C	Controlled cool to 65°C
Processing Time: 4-5 hours	Processing Time: 2+ hours

E-type thermocouples were used throughout and control was manual, a point to emphasize. The vacuum was provided by a good mechanical pump which could be isolated from the system, but the vacuum was pulled continuously throughout each run. A prerun requirement was that the evacuated bag/layup be isolated from the pump and hold vacuum at least momentarily before leaking slowly. The bag itself was usually of Kapton. Initial debulking was limited to only about 5 minutes. No effort was made to control resin bleed, nor was a dam used around the layup. The overall layup was wrapped with a bleeder ply to protect the vacuum bag. Teflon separator plies were placed between the prepreg and caul plate.

The 450-kHz constant frequency magnetic field was supplied by a Cycle-Dyne low voltage, high current, 3-kW power generation unit. This is not a portable device, but it is a workhorse and did provide a reliable and constant element. Two portable induction heating units were also investigated: the Model T-1000 and T-4000 systems

from Inductron. The T-1000 was a very early model, and this report will show only the results with the T-4000. These are portable 2-kW units, operating from 110V/20A supply and generate a variable frequency. The exact frequency depends on several factors including the "load." A working frequency of approximately 25 kHz was estimated for this effort. These units became available second-hand from another Air Force-sponsored contract and were in a used condition. They did, however, demonstrate the potential of portability for field repair. The 25-kHz frequency is not sufficient to significantly couple the small diameter carbon fibers for composite processing (assuming that to be mechanism dependent). Both of the composite materials studied could be processed with these units when susceptible caul plate materials were used.

A caul plate is a sheet of metal placed over the composite layup to effect equal pressure distribution and flatness of surface. Such procedure is standard with autoclave curing and can probably be used widely in the field. It is significant for induction heating that this caul plate material can be magnetically susceptible and/or electrically conductive. It can also be placed inside the vacuum bag to eliminate significant corrosion attack and can be sufficiently thin to conform to many surfaces under the influence of vacuum bagging. The following caul plate materials were studied in this effort: quartz and vycor (neither conductive nor magnetic), aluminum and copper (conductive and essentially nonmagnetic), steel (conductive and magnetic) and Admu 80, a commercial nickel-based alloy of very high magnetic susceptibility, approximately 350,000.

The use of caul plate materials with high magnetic susceptibility gives a different interpretation to the induction heating of composites. Steel and Admu 80, as used in this work, coupled very well to both 25-kHz and 450-kHz frequencies and provided a substantial source of heat to the underlying composite layup. Presumably this can be attributed to their I^2R losses, but they also serve as a magnetic shield to the layup. We did not make flux density measurements in this regard, but depending upon the degree of saturation, only some or none of the generated flux will actually get to the part when a magnetically susceptible caul plate is placed between it and the field. The results when using steel and Admu 80 as caul plates are thermally overwhelming compared to the other materials studied, so much so in fact that 0° thermoplastic laminates can be easily processed, a point worth emphasizing. Significantly less power is required for equivalent heating with magnetically susceptible caul plates, but induction heating in this vein assumes an altogether different quality. The use of variable caul plate materials also adds

another potential for study in that caul plate design, material selection, even more than one material might be used, and customized location become relevant.

Most of our work with the 450-kHz field used a pancake type coil. It was fabricated of 4.8-mm copper tubing, water cooled, and was approximately 15.2 cm in diameter with the leads extending vertically upward from the center and outer edge. It was not professionally made, and while it served very well, there were some areas of asymmetry in the winding. Flat specimens were processed directly under the coil, the extent of the effective range of the field being approximately 3.8 cm. The distance of separation between coil and sample, a critical variable, was measured from the top of the coil to the top of the vacuum bag. The Inductron unit most often used, the T-4000, was provided with its own coil, also of the pancake type, but it was not water cooled.

Coil design is one of the major aspects of induction heating, a vital element to determine distribution of the magnetic flux. Field repair will impose its own restrictions on coil geometry, and at least for now, a pancake shaped coil is required. One of the concerns with the pancake coil is that the field should be more dense around the perimeter because it is propagated spherically into space, and there are no cancellation effects on the outside of the outer coil turn. Measurements were not made in this regard, but temperature differences in the layup, from the outside in were always observable. At times these thermal differences appeared significant, reaching maximum differentials of 90°C in the 10.2-cm x 10.2-cm, 16-ply volume. Five thermocouples were usually imbedded throughout the layup, and temperature variations were consistently noted both in-plane and in-depth, although they were more generally around a 30°C range. A point to make regarding thermal differences is that they tend to increase as the temperatures themselves increase. That is, once a temperature difference is established, it will increase during the run, reflecting perhaps the changing conductivity of the carbon fibers. Both Miller (4) and Gillespie (5) comment on these temperature differentials, the latter maintaining that "heating is not uniform in such composites despite a uniform magnetic flux." These thermal differentials will have to be considered further if induction heating becomes more of a candidate for field repair.

A point of significant difference was observed between the 25-kHz and 450-kHz fields. Both fields provided immediate and thorough heating, given the prior comments regarding thermal differences. Both power supplies behaved this way with or without the use of susceptible caul plates; although without the susceptible caul plate, the response to 25 kHz is quite low. When the power was turned off with the 450-kHz field, cooling

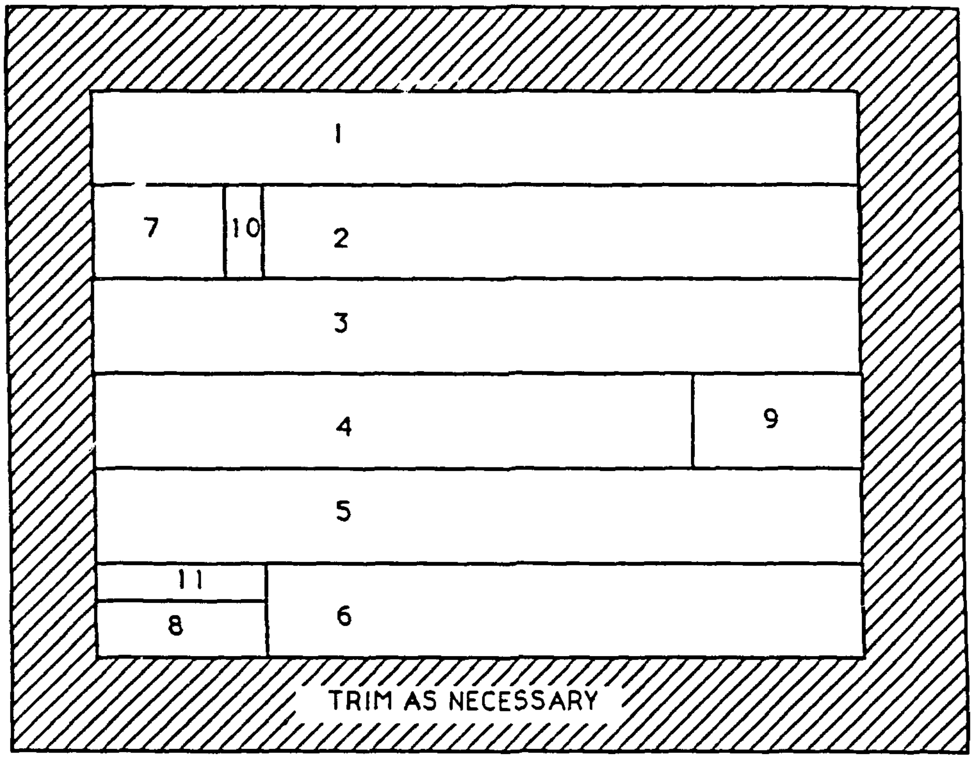
was immediate; in fact, the field had to be used to maintain a controlled cooling rate. There was a continuing increase in temperature when the lower frequency field was turned off. It is not clear if this was a field- or unit-related phenomenon. Also, reheating a cured or consolidated panel is not only immediate but occurs at a fraction of the power required to initiate heating the prepreg.

The sample size prepared in this work necessitated some specialized testing. The diagram in Table 2 illustrates how test samples were taken from the cured laminate. Photomicrographs were taken in orthogonal directions, and three nonneighboring samples were used for resin/fiber/void determinations. Mechanical strength tests were performed on the specimen sizes as noted in Table 2. Span/depth ratios of 32:1 and 16:1 were maintained for the flexure and shear tests respectively. Similar layups were processed under the normal autoclave cycles and also tested according to the plan in Table 2. These samples provided the standards for comparison.

TABLE 2

TEST PANEL LAYOUT

10.2 cm x 10.2 cm



1,3,5	3-PT FLEXURE (32:1)	8.26 cm x 1.0 cm (3.25" x 0.4")
2,4,6	4-PT SHEAR (16:1)	6.35 cm x 1.0 cm (2.5" x 0.4")
7,8,9	RESIN, FIBER, VOID (HNO_3 DIGESTION)	
10,11	PHOTOMICROGRAPHS, VOID	

SECTION 3
IM-6/3501-6

This material processed very well under the influence of the 450-kHz magnetic field with any type of caul plate. To elaborate more on the mechanism involved, note that once the prepreg was cured, reheating, again with any type of caul plate, required much less power.

The 450-kHz frequency enabled several variations to be studied regarding thermal rates and holds, peak cure temperatures, and times. Typical data are presented in Table 3 (physical properties), Table 4 (mechanical properties), and Table 5 (processing conditions) for 16-ply, quasi-isotropic layups of this system. The first listed specimen, A/C, is the autoclaved standard. The induction processed samples are ordered into two groups of five each. The first set, numbers 2, 3, 5, 6, and 7, were processed under a quartz caul plate. Here the heating mechanism involved the direct interaction of the prepreg and the magnetic field. The other set, numbers 1, 4, 8, 9, and 10 were processed under an Admu 80 caul plate. We might repeat that this program was introductory in nature; the data values are not intended to be optimum.

When processing a composite under vacuum bagging, the greatest concern becomes that of void content. It is the most sensitive and influential parameter to begin with, and because of the lower pressure, some higher levels of void concentration seem inevitable. The values in Table 3 were determined by the standard ASTM method of acid digestion using specific gravity values of 1.26 for the resin and 1.73 for the fiber. Values of perhaps 2 percent or less are encouraging but also suspect. Photomicrographic analysis indicates the values of Table 3 to be in error, yet the photographically measured values themselves show very poor precision. For example, void contents for four of these samples are compared in Table 6 under the different methods of analyses. The photomicrographic method of analysis consists of laying a gridwork of 266 intersecting points over a 50X photo that is 5.08 cm x 7.62 cm (2 in x 3 in). A representative sample surface must be selected, a decision that is never without question. If a point of intersection falls on a void the void is counted, otherwise it is not. This is a practical but nonstandard method, and a review of Table 6 indicates problems in picking the view, in relation to the fiber direction, as well as in selecting the specific surface area. The only summary comment to make now is that voids under vacuum bag processing are to be

TABLE 3

PHYSICAL PROPERTIES DATA; IM-6/3501-6
QUASI-ISOTROPIC, 16 PLY

No.	Specific Gravity	Voids Vol. %	Resin Wt. %	Fiber Vol. %	Thickness mm	Standard Deviation
A/C	1.58	0	31.1	62.9	2.144	0.0051
2	1.55	0.8	28.5	64.1	2.134	0.0229
3	1.54	0.8	30.0	62.8	2.100	0.0254
5	1.54	2.0	27.2	65.1	2.052	0.0305
6	1.54	2.5	25.9	65.6	2.032	0.0203
7	1.53	2.6	26.6	65.1	2.017	0.0305
1	1.55	0	30.6	62.4	2.271	0.0051
4	1.52	1.1	34.5	57.4	2.598	0.0991
8	1.48	3.2	35.0	55.8	2.408	0.0203
9	1.48	3.4	34.9	55.7	2.479	0.0508
10	1.48	3.6	34.7	55.8	2.464	0.0991

TABLE 4

MECHANICAL PROPERTIES DATA; IM-6/3501-6
QUASI-ISOTROPIC, 16 PLY

	3-Point Loading				4-Point Loading	
No.	Flexure Strength MPa	Std. Dev.	Modulus GPa	Std. Dev.	Interlaminar Shear Strength MPa	Std. Dev.
A/C	788.4	20.9	71.3	3.5	41.5	3.3
2	802.3	31.3	70.9	2.1	22.8	1.1
3	737.5	42.3	37.0	2.6	35.9	3.8
5	888.6	45.6	76.3	3.1	31.2	1.9
6	870.4	57.5	78.7	3.1	22.3	5.9
7	610.3	44.0	37.4	1.4	27.2	0.1
1	815.2	147.1	64.3	3.1	33.7	4.9
4	662.7	92.4	49.5	0.5	26.9	7.7
8	859.7	30.0	61.8	---	34.4	0.7
9	745.1	142.0	59.7	1.3	34.6	1.0
10	616.7	82.0	30.9	2.0	36.5	3.4

TABLE 5

THERMAL PROCESSING CYCLES
IM-6/3501-6, QUASI-ISOTROPIC, 16 PLY

No.	Ramp Rate °C/min	Time at 121°C	Total Time at 179°C	Processing Time (min)	Cured T_g
A/C	2.2	1 hr.	2 hrs.	251	146.7°C
2	5.6	30 min.	1 hr.	118	169.6°C
3	22.2	30 min.	1 hr.	97	145.8°C
5	5.6	10 min.	1 hr.	98	154.9°C
6	2.2	2.5 hrs.	2 hrs.	341	195°C
7	5.6	30 min.	1 hr.	118	181.3°C
1	5.6	None	30 min.	58	---
4	22.2	None	30 min.	37	---
8	27.8	None	30 min.	36	---
9	22.2	None	1 hr.	67	---
10	5.6	None	30 min.	58	---

TABLE 6

COMPARISON OF VOID CONTENT RESULTS
IM-6/3501-6, QUASI-ISOTROPIC, 16 PLY

No.	Digestion %	Photomicrographic View Void Determination Along		
		0° Plies (Only)	+45, -45 Plies (Only)	All Plies, %
2	0.8	---	---	4
5	2.0	7.6	5.1	7.6
6	2.5	5.6	5.3	8.5
7	2.6	7.6	6.8	11.3

expected, probably more than desired. Their presence is perhaps better measured in effect by variations in physical/mechanical properties, rather than in some absolute sense.

The specific gravity of the samples in Table 3 decline generally with increasing void content. This would be expected, but we must point out that all of those samples processed by induction heating show a specific gravity less than that of the autoclaved sample.

No real effort existed in layup or in processing to control bleed or resin content in the final laminate. The vacuum bag was transparent and the first evidence of bleed was observed at 65°C. A single bleeder ply was wrapped around the entire layup primarily to protect the vacuum bag. Teflon release plies were used between the prepreg and caul plate. Neither the resin nor fiber data of Table 3 correlate in any obvious manner to other parameters except the resin content is inversely related to processing time (Tables 3 and 5). Possible influence or control on final resin content is not a parameter to ignore, and any future work will bring it into consideration.

The panel thickness values in Table 3 present a point of interest in that some are actually less than that of the autoclaved sample [despite a difference in processing pressure between 483 kPa (autoclave) and 101 kPa (approximately 14.7 psi)]. The thickness values shown are the average of six determinations taken across the finished panel. For what it may be worth, and acknowledging that 10 samples do not make a strong statistical inference, the thinner samples (2,3,5,6, and 7) were all processed using a quartz caul plate, and the five thicker (than the autoclaved samples, 1,4,8,9, and 10) panels were processed using an Admu 80 caul plate. On the other hand, more realistically, the thinner samples were also those that had longer processing times, although the correlation here is not exactly one-to-one. In most cases, the longer processing times via induction heating were not as long as the autoclave cycle. The differences in standard deviation here are also notable. The value for the autoclaved sample is smaller in every case, and generally significantly smaller, than the vacuum bagged samples. This may indicate a degree of dimensional uniformity that is lost under the lower pressure process.

Table 4 presents the mechanical property values from 3-point loading (flexure), and 4-point loading (interlaminar shear). Each value represents the average of three samples per panel according to the diagram of Table 2. Some of the flexure values, both in magnitude and in standard deviation, are comparable to that of the autoclaved sample.

Also encouraging, the failure modes in flexure were predominantly compression/tension. Only occasionally was delamination observed. The interlaminar shear values, however, of the induction processed/vacuum bagged samples were never observed to be equivalent to those of the autoclave standard. The maximum here was about 80 percent of the autoclaved standard, but the failure modes were consistently that of interlaminar shear.

The thermal analysis data accumulated from these samples, Table 5, provided an approximate check on degree of cure and the glass transition temperature (T_g). There were no surprises but additional work must be done before conclusions are warranted. There is no reason why a particular degree of curing cannot be achieved.

Some (0,90), 16-ply, lay-up orientations were also processed from IM-6/3501-6. The overall property results followed the same patterns of the quasi-isotropic data in Tables 3 through 5. One notable difference was that the failure mode from flexure and shear testing often included delamination. This is neither surprising nor discouraging as (0,90) layups more frequently respond in this fashion.

The processing of IM-6/3501-6 (0,90) lay-up orientations also included the use of higher temperatures and additional variations in thermal ramp rates. Three sets of these panels were studied. Their properties are shown in Tables 7 and 8, and their processing variables, in Table 9. All of these samples are 16 ply. There was no intent to compare these values to the quasi-isotropic orientation; the purpose here was to investigate rapid processing.

The trade-offs in performance versus higher temperature/shorter time processing are evident in Tables 7, 8, and 9. We do not have criteria, per se, for a field patch other than it should "restore structural integrity," but no one expects the vacuum bagged material to be comparable to that from the autoclave, at least not on a one-to-one basis. Many of the operational constraints in the field have been mentioned, but another one that can be present is that of available time. In an emergency, the patching technique must not only be "acceptable" but also rapid. Tables 7 through 9 begin to show that induction heating as field repair technique can potentially meet short time processing constraints. Whether or not the composite will be acceptable in a full structural integrity restoration sense must still be decided.

TABLE 7

PHYSICAL PROPERTIES DATA; IM-6/3501-6
(0,90) LAYUP, 16 PLY

No.	Resin Wt. %	Fiber Vol. %	Specific Gravity	% Voids (Digestion)	Thickness mm	Standard Deviation
A/C	32.2	62.3	1.59	0	2.134	0.0178
17	29.9	61.8	1.52	2.0	2.316	0.0406
19	28.3	63.4	1.53	2.2	2.670	0.1067
18	27.9	63.7	1.53	2.4	2.235	0.0076
I2	29.7	62.9	1.55	0.6	2.243	0.0178
I3	29.1	63.9	1.56	0.2	2.375	0.0330
I4	28.3	65.1	1.57	0	2.212	0.0152
5	34.8	55.9	1.48	3.0	2.535	0.0889
6	32.2	59.8	1.53	1.2	2.525	0.0584
4	30.0	63.5	1.57	0	2.398	0.1067

TABLE 8

MECHANICAL PROPERTIES DATA; IM-6/3501-6
(0,90) LAYUP, 16 PLY

	3-Point Loading				4-Point Loading	
No.	Flexure Strength MPa	Std. Dev. MPa	Modulus GPa	Std. Dev. MPa	Interlaminar Shear Strength MPa	Std. Dev. MPa
A/C	1164.0	104.9	66.2	1.3	55.2	2.8
17	1074.3	51.8	71.1	22.1	45.5	2.8
19	865.3	133.9	67.6	1.4	28.3	0.7
18	845.2	16.6	67.6	1.4	47.6	1.4
I2	900.4	23.5	69.0	0.3	24.2	0.7
I3	773.5	66.9	69.0	0.3	24.8	2.1
I4	1042.6	91.1	80.0	1.9	27.6	2.8
5	551.3	180.8	75.9	0.6	24.8	1.4
6	712.1	75.2	72.4	3.7	28.3	2.1
4	1012.2	111.8	60.0	47.6	37.3	8.3

TABLE 9

THERMAL PROCESSING CYCLES
IM-6/3501-6, (0,90) LAYUP, 16 PLY

No.	Ramp Rate °C/min	Time @ 121°C min	Time @ Cure Temp. hr.	Total Processing Time (min)
A/C	---	---	---	251
17	6	None	199°C/1	92
19	6	15	199°C/0.5	77
18	6	15	199°C/1	107
I2	6	30	182°C/1	119
I3	6	None	182°C/1	89
I4	20	20	182°C/0.5	58
5	Thermal spikes: 28°C/min, 5 min cool, overall @ 6°C/min		193°C/1	---
6	---	40	193°C/1	68
4	Thermal spikes: 56°C/min, 10 min cool, overall @ 2°C/min		199°C/1	---

SECTION 4
IM-6/PEEK

4.1 THE 10.2-cm x 10.2-cm LAMINATES

The processing of thermoplastic composites requires heating the resin to the melt temperature, holding for a time under pressure, and cooling. There is no chemistry involved, the material is softened, it conforms under the effects of temperature and pressure, and retains its shape upon cooling.

The change in resin, from the 3501-6 thermoset to the thermoplastic PEEK, did not appear to have measurable effects on the techniques and requirements of induction heating. The 450-kHz field can raise the IM-6/PEEK to processing temperature (cf Table 1). But one observation should be noted.

As this program continued a lessening of response to the 450-kHz field was noted from the thermoplastic material when the quartz caul plates were used. The prepreg had been stored at room temperature for a lengthy period (over 3 years), and the lower response with storage time is attributed to some degree of "set" taken by the resin, which then insulated the plies more effectively. This effect might be noticeable only under the pressure limitations of vacuum bagging. It could not be predicted and was only observed when the quartz caul plates were used. Typically, with the older resin/prepreg and quartz caul plate in place, higher power levels became necessary for the same material as the program went on. But at temperatures around 210°C, the part temperature would suddenly and literally shoot upward so rapidly, in fact, that the runs had to be aborted. This phenomenon might be attributed to sudden contact of the fibers owing to resin softening with temperature. It becomes something of a material storage problem that will have to be addressed in more detail as induction heating becomes more applicable to composite materials. Another related point that can be reiterated here is the rapidity of temperature increase that is possible with induction heating under the conditions described. Thermal rates of several hundred degrees per minute are available.

The property data for the thermoplastic PEEK of $(0,90)_{4S}$ layup are shown in Tables 10 and 11. All of these data are based on the test sample schematic of Table 2. Two autoclaved standards are included with the IM-6 fiber, and one with AS-4 fiber for comparison. The induction heated samples, numbers I10 and I13, were processed under an Admu 80 caul plate; thus these are results from susceptor heating. The higher strength

TABLE 10

PHYSICAL PROPERTIES DATA, IM-6/PEEK
(0,90) LAYUP, 16 PLY

No.	Specific Gravity	Resin Wt. %	Fiber Vol. %	Voids Vol. %	Thickness mm	Standard Deviation
A/C 1*	1.57	31.6	62.1	0	2.235	0.0508
A/C 2	1.57	31.8	61.4	1.2	2.347	0.0229
I10	1.56	31.1	62.0	1.1	2.332	0.0483
I13	1.55	33.0	60.0	1.5	2.433	0.0356
A/C 3	1.60	32.3	60.2	0.8	2.113	0.0152

* First four panels were IM-6/PEEK; A/C 3 was AS-4/PEEK.

TABLE 11

MECHANICAL PROPERTIES DATA, IM-6/PEEK
(0,90) LAYUP, 16 PLY

	3-Point Loading				4-Point Loading	
No.	Flexure Strength MPa	Standard Deviation	Modulus GPa	Standard Deviation	Interlam. Shear Str. MPa	Standard Deviation
A/C 1*	1125.4	30.4	90.4	4.8	37.3	1.4
A/C 2	1130.9	73.1	89.7	3.4	64.2	19.3
I10	972.2	16.5	78.7	3.4	66.9	0.7
I13	908.7	179.4	84.9	2.8	68.3	12.4
A/C 3	1193.0	47.6	73.8	0.7	69.0	0.7

* First four panels were IM-6/PEEK; A/C 3 was AS-4/PEEK.

levels of the thermoplastic over the thermoset materials are evident, and there is also a point to note regarding the void contents. The void concentrations are lower with the thermoplastic and agreement is better between the digestion and photographic methods for the thermoplastic material than was the case for the thermoset situation (as discussed earlier). Also, the interlaminar shear values of the vacuum bagged material equal those from the autoclaved material, but the 3-point flexure values are somewhat lower. The low value of interlaminar shear for sample number A/C 1 is regarded as an anomaly.

We also successfully processed a 10.2-cm x 10.2-cm, 16-ply IM-6/PEEK panel within a total processing time of 15 minutes - 5 minutes to 382°C; 5-minute hold; 5-minute cool down. An identical processing schedule was successfully applied to a similar panel of the same overall size but whose plies were composed of 5.1-cm x 10.2-cm sections which formed together with processing. These were strictly experimental runs to gain short time processing experience with the thermoplastic material. No physical or mechanical evaluations were conducted.

These data and experiments are not yet sufficient to warrant firm conclusions, but there does not appear to be a major reason why induction heating cannot be adapted to thermoplastic repair.

4.2 THE 5-cm x 5-cm LAMINATES

The IM-6/PEEK system was also studied using a smaller sized induction coil. A 7.62-cm diameter coil, similar in other respects to the 15.24-cm-diameter coil, was used to prepare 16-ply $(0,90)_{4S}$ panels, 5.08 cm x 5.08 cm. Autoclaved samples were also used here as standards. The induction heated samples were processed under an aluminum caul plate. The usual physical properties were measured (three test pieces per panel), but the mechanical property evaluation consisted of a short beam shear (SBS) test on two test coupons per panel. These results are shown in Tables 12 and 13. The SBS results reported in these tables are based upon two panels, four test specimens for the autoclaved material, and on one panel, two test specimens for those which were induction heated. The induction heating processing cycles were varied as noted in Table 13.

The mechanical property results, SBS, of the vacuum bagged samples are identical with those from the autoclave, but the physical property differences require further study. The lower specific gravities of the vacuum bagged materials correlate to higher void contents, but there is a 3- to 4-percent difference in resin content, and a

7-percent difference in fiber content, yet the vacuum bagged samples for the most part are thinner than those from the autoclave.

TABLE 12
PHYSICAL PROPERTIES DATA, IM-6/PEEK
(0,90) LAYUP, 16 PLY
5-CM x 5-CM LAMINATES

No.	Specific Gravity	Void Vol. %	Resin Wt. %	Fiber Vol. %	Thickness mm	Standard Deviation
A/C	1.62	0	28.5	66.95	2.238	0.1372
4-12	1.55	1.3	32.6	60.4	2.235	0.0356
4-10	1.55	1.4	32.8	60.1	2.197	0.0178
4-11	1.55	1.6	31.9	60.7	2.144	0.0483
4-18	1.54	1.8	32.2	60.5	2.225	0.0152
4-17	1.54	2.0	31.2	61.4	2.268	0.0203

TABLE 13
MECHANICAL PROPERTIES DATA, IM-6/PEEK
(0,90) LAYUP, 16 PLY
5-CM x 5-CM LAMINATES

No.	Short Beam Shear Strength MPa	Standard Deviation	Processing
A/C	73.1	0.34	per Table 1
4-12	73.8	0.0	Slow heat; 2°C/m; 25m/382°C
4-10	68.3	2.1	4.5°C/m; 30m/382°C (Normal)
4-11	74.5	2.1	Several rapid high Temp spikes/ cool, to 30m/382°C
4-18	73.8	2.1	11°C/m; 30m/382°C
4-17	79.4	1.4	Long debulk; 45m/393°C

SECTION 5
CAUL PLATE MATERIALS

There are several ways of illustrating the thermal effects of the different materials used as caul plates in this work. In general, the following can be observed:

1. The vycor or quartz (both were used) materials do not appear to interact with the magnetic field, and all of the heating comes from the field/sample interaction. It is possible with 450-kHz frequency to process both the thermoset and the thermoplastic materials, but power requirements are higher than when a caul plate/susceptor is used.

2. The results of using metallic caul plates depends first on the magnetic properties of the metal. Aluminum and copper are basically nonmagnetic (aluminum is slightly paramagnetic, copper is slightly diamagnetic) but both exhibit slight to moderate heating when interacting with the magnetic field. The use of metals with high magnetic susceptibilities, such as steel and Admu 80, provide a significant source of heat to the prepreg sample.

The sizes of the caul plate materials used in this work were all matched to the sample area. They had the following thicknesses: quartz/vycor (0.032 mm), copper (0.0071 mm), aluminum (0.0160 mm), steel (0.0094 mm), Admu 80 (0.0028 mm). A summary illustration of the relative effects of these various materials as caul plates may be seen from the following. Two 10.2-cm x 10.2-cm caul plates were taped closely together with five thermocouples along an inside diagonal. This alone constituted a test sample; there was no prepreg and no vacuum bagging. The samples were placed in the 450-kHz field, and similar power settings were used for all the materials. In equivalent time periods and at equal distances of coil/sample separation, the following temperatures were attained with each material.

Vycor	Copper	Aluminum	Steel	Admu 80
RT	27°C	28°C	116°C	149°C

To whatever extent the applied magnetic field reaches the sample when the various metals are used as caul plates is not known, but the results with steel and Admu 80 are clearly overwhelming to the "field-only" effects from quartz and vycor.

One other result can be mentioned in this regard. Some preliminary work indicated that stacking the Admu 80 caul plates in the field but outside the vacuum bag tended to provide significantly improved temperature equilibrium within the sample, i.e. the overall thermal gradients were much less. The caul plates become very hot and the practicality of the practice can be questioned, but something in the way of less power/lower field intensity, greater field uniformity, or other phenomena resulted from this stacking to improve thermal distribution within the sample.

SECTION 6
USE OF COIL VIBRATION

Coil design is yet to be addressed with the demands of field repair, but given a pancake type coil, one method to offset the nonuniformities of applied field or sample temperature differentials might be to vibrate the coil across the sample during the processing. Relative motion is what is desired; the sample could also be moved. We noticed that even slight movement of the sample by hand under the coil resulted in immediate temperature fluctuations which seemed to indicate the thermal dynamics within the sample. To study coil movement, a small vibration motor was mounted on a flexible strip of plastic: one end of the strip was secured and the coil leads were attached to the other end. The motor was operated from a variable voltage supply and transferred its vibrational movement to the coil. This was not altogether quantitative nor was the procedure optimized, but a distinct improvement toward temperature uniformity within the sample was noted. Typical results are shown in Table 14. These data are for a thermoplastic processing run; the overall sample temperature is increasing to 382°C. The coil was vibrated rapidly across the sample with about a 1-inch amplitude. The temperature differentials within the sample increase as a function of overall temperature.

TABLE 14
EFFECT OF COIL VIBRATION ON
MAXIMUM TEMPERATURE GRADIENTS
(5 thermocouples throughout sample)

With Vibration	Without Vibration	
12°C	18°C	Data are for thermoplastic processing run. Overall temperature is increasing down the column to 382°C.
27°C	23°C	
31°C	44°C	
37°C	49°C	
39°C	55°C	
40°C	56°C	
42°C	58°C	
43°C	59°C	
43°C	60°C	

SECTION 7
OTHER LAYUPS

Whatever the mechanism of energy transfer from the magnetic field to the organic composite, the lay-up orientation of the prepreg sample clearly exerts a significant influence. A zero degree orientation could not be heated, at least not with our unit at maximum power and minimum distance of coil/sample separation. As the plies were crossed and varied from 0° to 30°, to 45°, to 90° in orientation, the power required to heat to a specific temperature at a constant distance of coil/sample separation became progressively less. This relationship between the distance of separation, the power setting, and the sample layup is shown in a relative way in Table 15.

Further pointing out the importance of ply orientation was the fact that layups of $(0,0,90,90)_{4S}$ and $(0,90)_{8S}$ of IM-6/PEEK could both be processed under an Admu 80 caul plate at less power than a zero-degree, 16-ply sample required. This may be attributed to the effect of the crossed plies. But this result also implies that the Admu 80 caul plate (which is not grounded) may be saturated and some flux may be getting to the sample directly, either through or around the caul plate.

We attempted to process a two-ply, (0,90) layup of IM-6/PEEK using a quartz caul plate but could only observe very slight heating at maximum power and minimum distance of coil/sample separation. Four plies, $(0,90)_{4S}$, did heat slowly, and gained about 20 degrees in temperature. But this rise was observed only at the edges of the sample. The center portion did not heat at all. This emphasizes the perimeter/interior thermal differentials discussed earlier, and the need for coil design to (maybe) offset these temperature differences that appear to be caused by a nonuniform field density. The lack of thermal conductivity within the sample is also evident. In fact, the lack of or low thermal conductivity of the composite was observed many times as samples could be held under constant power settings and measurable temperature differentials for long periods without any indication of realizing an equilibrium of temperature.

It is worth noting that vacuum bagging is necessary to get any response at all from the sample. Any number of plies (up to 16), however oriented, will not excite even under maximum power unless pressed together, by vacuum bagging in this case.

TABLE 15

RELATIVE COMPARISON OF COIL/SAMPLE DISTANCE, LAYUP ORIENTATION, AND POWER REQUIRED

Distance and Separation, Coil and Sample	Sample Layup Orientation	Power Required
Minimum	0°	Will not heat
Minimum	$(0,30)_{4S}$	High
Moderate		Very High
Maximum		Maximum
Minimum	$(0,45)_{4S}$	Moderate
Moderate		High
Maximum		Maximum
Minimum	$(0,90)_{4S}$	Minimum
Moderate		Minimum
Maximum		Moderate
Minimum	Quasi-isotropic 16 ply	Minimum
Moderate		Minimum
Maximum		Moderate

To emphasize again the effects of the Admu 80 caul plate, a two-ply, zero-degree sample of IM-6/PEEK was readily processed and gave the following properties: specific gravity, 1.55; 33.1 percent resin; 59.9 percent fiber; 1.3 percent voids.

Other sample thicknesses that were successfully processed include IM-6/3501-6 samples, (0,90), of 32 plies and 125 plies. Both of these layups were processed under a quartz caul plate. The 125-ply sample gave the following analytical results: 1.50 specific gravity; 34.0 percent resin; 57.3 percent fiber; 2.17 percent voids. We made only one run with this thickness of sample; it is not possible to comment on any thermal exotherming during processing.

SECTION 8
CONCLUSIONS

The IM-6/3501-6 and IM-6/PEEK prepreg materials respond readily to an alternating magnetic field of 450 kHz. The rate of temperature rise is immediate and is directly proportional to the power applied, inversely proportional to the distance of coil/sample separation, and directly related to the ply orientation. Either vacuum bagging or some other form of consolidation pressure is necessary to effect this response.

These prepreg materials show minimal response to a field of approximately 25 kHz. But the use of particular metals as caul plate/susceptors will allow the processing of these prepregs at this lower frequency, although the mechanism of heating now becomes more "traditional." The advantage of the low frequency is portability (and cooling water is not required).

There is perhaps an inherent temperature differential across the prepreg sample during processing with the pancake type induction coil. In general the portions of the sample under the periphery of the coil become hotter than the interior. The use of a susceptor/caul plate skews this thermal gradient, the temperature uniformity of the sample is improved and there is no repeatable pattern of thermal gradients from sample to sample.

Processing these composites under vacuum bagging consolidation provides a different set of results as related to the thermoset vs. the thermoplastic materials. The void content is more critical with the thermoset; in general, the void levels are higher with vacuum bagging as opposed to the autoclave. This is not surprising, and any property comparisons or analysis should be done with this in mind. The interlaminar shear strengths, 4-point loading, are about 75-80 percent of the autoclaved material, but the flexure strengths, 3-point loading, can be equal to autoclaved material. Void content is less of a problem with thermoplastic processing, but here it is the shear strengths that are comparable to the autoclaved samples, whereas the flexure strength is about 85 percent of the standard. These overall conclusions are not intended to be generalized but are based on the work herein.

Induction heating does present the possibility of reduced processing time and this can be an advantage in the field. The use of a caul plate/susceptor adds further to this potential. Rapid and controlled temperature profiles are possible, and portability becomes a reality, at least with a caul plate/susceptor.

SECTION 9
REFERENCES

1. C. A. Tudbury, Basics of Induction Heating, John F. Rider, Inc., New York, 1960, pp. 1-13.

2. S. Zinn and S. L. Semiatin, Elements of Induction Heating, Electric Power Research Institute, Palo Alto, 1988, pp. 9.

3. J. Border, "Understanding Induction Heating and Its Utilization for Aircraft Structural Repair," Final Report for McDonnell Aircraft Company Contract No. F33657-88-C-0087, February 1989.

4. A. K. Miller, et al., SAMPE J., 26, 4 (1990).

5. B. K. Fink, R. L. McCullough, J. W. Gillespie, "A Local Theory of Heating in Cross-Ply Carbon Fiber Thermoplastic Composites by Magnetic Induction," CCM Report 90-37, University of Delaware.

6. ibid., "A Model to Predict the Planar Electrical Potential Distribution in Cross-Ply Carbon-Fiber Composites Subjected to Alternating Magnetic Fields," CCM Report 91-57, University of Delaware.

7. ibid., "A Model to Predict the Through-Thickness Distribution of Heat Generation in Cross-Ply Carbon-Fiber Composites Subjected to Alternating Magnetic Field," CCM Report 91-58, University of Delaware.

8. ibid., "The Influence of Moisture on Magnetically Induced Dielectric Heating of Poly-Etheretherketone/Carbon Fiber Composites," CCM Report 91-59, University of Delaware.

9. ibid., "Experimental Verification of Models for Induction Heating of Continuous-Carbon-Fiber Composites," CCM Report 91-60.

10. J. Border and R. Salas, SAMPE International Symposium, 34, 2569 (1989).

11. W. Lin, O. Buneman, A. K. Miller, "Induction Heating Model for Graphite Fiber/Thermoplastic Matrix Composites," SAMPE J., 27, 6 (1991).

12. J. W. Sobczak. U.S. Army Aviation Systems Command, Fort Eustis, VA, Private Communication.

U.S. Government Printing Office 550-106